**B. OLEKSIAK, G. SIWIEC**

ISSN 0543-5846
METABK 51(1) 25-27 (2012)
UDC – UDK 669.2/:669.4:669.3:621.694:546.293=111

# REMOVAL OF LEAD FROM THE INDUSTRIAL AND SYNTHETIC Cu-Pb-Fe ALLOY WITH ARGON BARBOTAGE

Received – Prispjelo: 2011-01-12
Accepted – Prihvaćeno: 2011-03-28
Original Scientific Paper – Izvorni znanstveni rad

Results of research on removal of lead from synthetic and industrial Cu-Pb-Fe alloy with argon barbotage are presented. For examinations was taken a synthetic alloy and industrial alloy coming "Glogow II" Copperworks. As basic research equipment was used a pipe resistance furnace enabling heating of samples up to 1 473 K. Examinations were made in 2 test series. The 1 series was performed on the synthetic alloy, while in 2 series was used an industrial alloy. All series were conducted at 1 473 K and with gas flow $5{,}55 \cdot 10^{-6}$, $6{,}94 \cdot 10^{-6}$, $8{,}33 \cdot 10^{-6}$, $9{,}72 \cdot 10^{-6}$ $m^3 \cdot s^{-1}$.

*Key words:* metallurgy, barbotage, copper, refining, alloy

**Odstranjivanje olova iz industrijske i sintetičke Cu-Pb-Fe legure argonskim pročišćavanjem.** Daju se rezultati istraživanja odstranjivanja olova iz sintetičke i industrijske Cu-Pb-Fe legure argonskim pročišćavanjem. Za ispitivanje odabrana je sintetička industrijska legura iz tvrtke za bakar "Glogow II". Osnovni uređaj za istraživanje je cjevasta peć sa mogućnošću zagrijavanja uzoraka iznad 1 473 K. Ispitivanja su provedena u 2 serije. Prva serija je provedena na sintetičkoj leguri, a druga serija na industrijskim legurama. Sve serije su izvedene na 1 473 K sa strujom plina $5{,}55 \cdot 10^{-6}$, $6{,}94 \cdot 10^{-6}$, $8{,}33 \cdot 10^{-6}$, $9{,}72 \cdot 10^{-6}$ $m^3 \cdot s^{-1}$.

*Ključne riječi:* metalurgija, pročišćavanje, bakar, refinacija, legura

## INTRODUCTION

One of products of the single step copper smelting of concentrate in fluidized-bed furnace in Glogow Copperworks is the slag containing up to 20 % wt. copper. This material is then submitted to a decopperizing process in electric furnace. As a product in this technology the Cu-Pb-Fe alloy containing over 20 % wt. of lead and the dusts containing up to 40 % wt. of this element are obtained [1, 2].

Cu-Pb-Fe alloy is processed in the converter to obtain copper suitable for the fire refining process, i.e. containing below 0,3 % wt. of lead. At the same time, this technology is aimed to removal of possible highest quantity of lead in dusts and obtainment of low concentration of arsenic in copper. As result of this process, approx. 70 % wt. of lead contained in this alloy is passing to dusts, and the remaining part is collected in converter slag. Because the copper content in slag (even above 35 % wt.) is higher than that of lead, this material is returned back to electric furnace. It can be assumed then, that the lead contained in converter slag is circulating in the process cycle [3-6].

B. Oleksiak, G. Siwiec - Silesian Technical University, Department of Metallurgy, Katowice, Poland

## RESEARCH EQUIPMENT AND MATERIALS

The research work was made on synthetic alloy Cu-Pb-Fe and on industrial alloy provided by „Glogow" Copperworks. The synthetic alloy was manufactured from deoxidized copper M0B grade, lead PbO grade and Armco iron with melting of the metallic components together in vacuum-type induction furnace IS/III mark Lebold Heraus, in argon atmosphere and. in SiC crucible. The alloy was poured into preheated graphite moulds, to ingots in form of rods – 0,023 m dia. After extraction from moulds, the rods were cleaned and cut into cylindrical samples of 0,022 m dia. and h = 0,07 m. The chemical composition of alloys used in examinations is presented in Table 1.

Table 1 **Chemical composition of tested alloys**

| Type of alloy | Contents of alloy basic parts / % wt. | | | | | |
|---|---|---|---|---|---|---|
| | Cu | Pb | Fe | As | S | Ag |
| Synthetic | 74,8 | 22,5 | 4,6 | --- | --- | --- |
| Industrial | 70,4 | 21,5 | 2,9 | 3,4 | 1,7 | 0,0025 |

A sketch of the equipment used in tests is showed in Figure 1. The main component was a pipe-type resistance furnace, enabling heating of sample up to 1 473 K. In the furnace was placed a ceramic protective retort accommodating the crucible and the sample. The process temperature was measurmed with a thermocouple Pt-PtRh10 of 0,006 m dia. placed directly in liquid alloy. The gas was introduced into metallic bath through

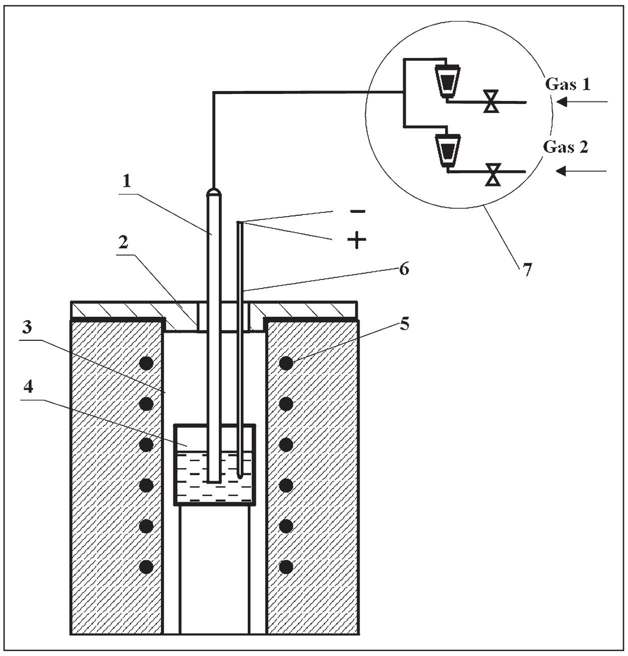


**Figure 1** Sketch of equipment used in tests: 1 - lance, 2 – asbestos seal, 3 – ceramic shield, 4 – crucible with alloy, 5 – furnace heating components, 6 – thermocouple, 7 – gas supply system.

an $Al_2O_3$ lance immersed to the depth 0,005 m to the crucible bottom. Gas flow was controlled by manual valves and rotameters. Whole system was closed with a ceramic cover and asbestos seal.

## METHODOLOGY OF RESEARCH

A prepared alloy sample put in alundum crucible was introduced into ceramic retort placed in the furnace heated to required temperature.

In the course of melting stage, an inert gas was introduced into working chamber through a lance located above crucible to the protect the metal against oxidation. When predetermined temperature was achieved in the furnace, a lance was introduced in and appropriate gas flow was adjusted. At the set time intervals the alloy specimens were sampled with quartz pipes. During this operation the flow of gas through metallic bath was uninterrupted, a possibility of adverse metal delamination was prevented. Having completed the tests, the lance was withdrawn off crucible, furnace supply was switched off and the system was cooled down with inert gas blowing. After sample withdrawal, the slag and metallic fractions were separated and the samples were weighed up. In case of metallic samples, they were wholly dissolved in appropriate solution of nitric acid and the resultant solutions were analyzed with the help of atomic absorption spectrometer Unicam, model Solar M6.

## SCOPE OF RESEARCH

Examinations were carried out in 2 measurement series. The Ist series was executed with use of synthetic alloy, while the IInd series used an industrial alloy from "Glogow II" Copperworks. All series were conducted at of the temperature 1 473 K, with gas flow of $5{,}55\cdot10^{-6}$, $6{,}94\cdot10^{-6}$, $8{,}33\cdot10^{-6}$, $9{,}72\cdot10^{-6}\,m^3\cdot s^{-1}$. In Table 2 are shown the basic test parameters for measurement series.

Table 2 **Basic test parameters**

| Series No. | Alloy type | Applied gas | Test duration / s | Sample weight /g |
|---|---|---|---|---|
| I | synthetic | Ar | 10800 | 490±10 |
| II | industrial | Ar | 10800 | 490±10 |

Measurement series are designated as follows: alloy type – process temperature– first three figures define the gas flow. For example, the test designation S-1 473-5,55-1 means, that this examination is related to the first measurement series executed at 1 473 K temperature, with argon flow $5{,}55\cdot10^{-6}\,m^3\cdot s^{-1}$, test 1.

## TEST RESULTS

Test results of all experiments concerning final concentration of lead and its degree of removal in liquid Cu-Pb-Fe alloy are presented in Table 3. In Figure 2 and 3 are shown exemplary variations of lead concentration depending on process duration achieved in experiments series 1 and 2.

Table 3 **Final concentrations of lead and its elimination degree achieved in tests series 1**

| No. | Test designation | Final concentration of lead in alloy / % wt. | Lead removal degree / % wt. |
|---|---|---|---|
| 1 | S-1473-5,55-1 | 21,62 | 3,91 |
| 2 | S-1473-5,55-2 | 21,64 | 3,82 |
| 3 | S-1473-6,94-1 | 21,42 | 4,79 |
| 4 | S-1473-6,94-2 | 21,46 | 4,62 |
| 5 | S-1473-8,33-1 | 21,23 | 5,65 |
| 6 | S-1473-8,33-2 | 2,.33 | 5,21 |
| 7 | S-1473-9,72-1 | 21,24 | 5,59 |
| 8 | S-1473-9,72-2 | 21,04 | 6,48 |
| 9 | P-1473-5,55-1 | 21,24 | 1,26 |
| 10 | P-1473-5,55-2 | 21,30 | 0,98 |
| 11 | P-1473-6,94-1 | 20,99 | 2,42 |
| 12 | P-1473-6,94-2 | 21,02 | 2,28 |
| 13 | P-1473-8,33-1 | 20,88 | 2,93 |
| 14 | P-1473-8,33-2 | 20,86 | 3,02 |
| 15 | P-1473-9,72-1 | 20,53 | 4,56 |
| 16 | P-1473-9,72-2 | 20,49 | 4,74 |

The variations of lead mole fraction in metal bath for test series 1 and 2 are correlated with the function:

$$X_{Pb} = A \cdot t + B \qquad (1)$$

with:

$X_{Pb}$ – lead mole fraction,

t – process duration time/ s

A – equation constant / $s^{-1}$

B – equation constant.

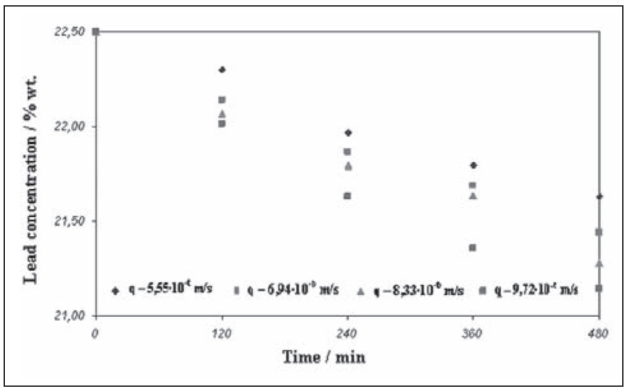


**Figure 2** Variation of lead concentration in Cu-Pb-Fe synthetic alloy depending on process time

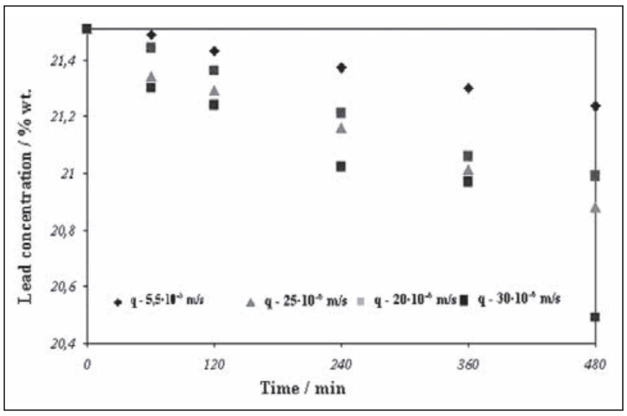


**Figure 3** Variation of lead concentration in Cu-Pb-Fe industrial alloy depending on process time

In Table 4 are presented exemple values of parameter A equation (1), its standard deviation and the values of correlation factor of obtained test results for test series 1.

Table 4 **Results of correlation of mole fraction changes of lead in Cu-Pb-Fe alloy in function of process duration time for test series 1**

| Test designation | $-A\cdot10^6$ /$s^{-1}$ | Standard deviation, /$S(A)\cdot10^6$ | Standard deviation /$S(B)\cdot10^4$ | Correlation factor / r |
|---|---|---|---|---|
| S-1473-5,55-1 | 0,097 | 0,006 | 1,16 | 0,986 |
| S -1473-5,55-2 | 0,110 | 0,015 | 2,78 | 0,942 |
| S -1473-6,94-1 | 0,120 | 0,007 | 1,38 | 0,987 |
| S -1473-6,94-2 | 0,117 | 0,010 | 1,78 | 0,978 |
| S -1473-8,33-1 | 0,137 | 0,012 | 2,05 | 0,978 |
| S -1473-8,33-2 | 0,127 | 0,012 | 2,17 | 0,972 |
| S -1473-9,72-1 | 0,139 | 0,017 | 2,97 | 0,958 |
| S -1473-9,72-2 | 0,172 | 0,014 | 2,53 | 0,979 |

## CONCLUSIONS

The results of tests and their analysis provide the grounds to formulate the following conclusions:

1. Increase of gas flow intensity and temperature of the process will raise the degree of lead removal from tested alloys.
2. Maximum lead removal degree at parameters used for tests was to 6,8 % wt for the synthetic alloy and 4,74 % wt for the industrial alloy.
3. Low speed of barbotage process would not allow to apply this technology as the first stage of converting Cu-Pb-Fe alloy. It is possible to use this technology as an auxiliary operation. Such operation can be applied in the second converting stage, when the lead content in copper is dropping to 2 %.

## REFERENCES

[1] A. Piestrzyński, A. Jasiński, J. Kotarski & others: Monograph KGHM Polska Miedź S.A., Cuprum Sp. z.o.o., Lublin (1996).

[2] M. Kucharski: Pyrometallurgy of Copper, Wyd. Nauk. – Dydakt., Kraków (2003).

[3] L. Blacha, J. Łabaj, B. Oleksiak: 12-th International Metallurgical and Materials Conference “Metal 2003”, Czech Republic (2003).

[4] B. Oleksiak: Metalurgija, 48 (2009)2, 87-90.

[5] H. S. Sohn, T. Fukunaka: Metall. Trans. B 35B (2004), 651.

[6] K. Okumara, M. Ban: ISIJ Int. 35(1995), 832.

**Note:** P. Nowak is responsible for English language, Katowice, Poland